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SPECIFICATION FOR BROMAMINE ACID, TECHNICAL

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Indian Standard
SPECIFICATION FOR BROMAMINE ACID, TECHNICAL

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(*Continued on page* 2)

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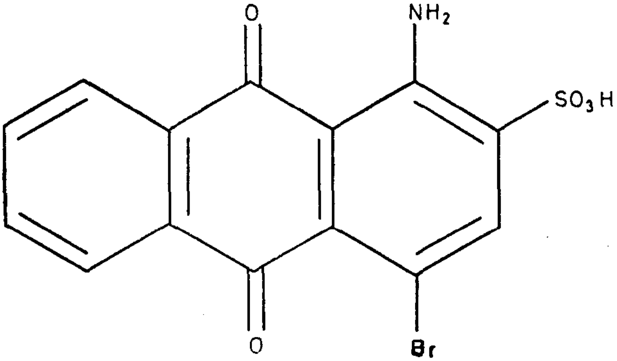
Indian Standard

SPECIFICATION FOR BROMAMINE ACID, TECHNICAL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 8 June 1978, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 Bromamine acid ($C_{14}H_8BrNO_5S$) is an important dye intermediate used in the manufacture of various types of dyes. It is also known as 1-amino-4-bromoanthraquinone-2-sulphonic acid. It has the following structural formula :



BROMAMINE ACID
(Molecular Mass 381·9)

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

---

*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for bromamine acid, technical.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of bright red powder. It shall be free from dust and other visible impurities.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR BROMAMINE ACID, TECHNICAL

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CL No. IN APPENDIX A)
(1)	(2)	(3)	(4)
i)	Assay, percent by mass, *Min*	85	A-1
ii)	Unsulphated matter, percent by mass, *Max*	2	A-2
iii)	1-Aminoanthraquinone-2-sulphonic acid, percent by mass, *Max*	3	A-3
iv)	Moisture, percent by mass, *Max*	5	A-4

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in steel drums (*see* IS : 2552-1970*) lined with suitable polyethylene film or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

3.2 Marking — Each container shall bear legibly and indelibly the following information:

a) Name of the material;
b) Name of the manufacturer and his recognized trade-mark, if any;
c) Batch number; and
d) Tare, net and gross mass.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

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*Specification for steel drums (galvanized and ungalvanized) (*first revision*).

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in **3** of IS : 5299-1969*.

4.2 Number of Tests

4.2.1 Test for assay shall be conducted on each of the individual samples.

4.2.2 Tests for the determination of the remaining characteristics given in Table 1 shall be conducted on the composite sample.

4.3 Criteria for Conformity

4.3.1 *For Individual Samples* — The lot shall be declared as conforming to the requirement of assay if each of the individual test results as obtained in **4.2.1** satisfies the relevant requirement given in Table 1.

4.3.2 *For Composite Sample* — The lot shall be declared as conforming to the requirements if the test results as obtained in **4.2.2** satisfy the corresponding requirements given in Table 1.

5. TEST METHODS

5.1 Tests shall be carried out according to the methods prescribed in Appendix A as indicated in col 4 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

(*Table* 1, and *Clause* 5.1)

METHODS OF TEST FOR BROMAMINE ACID, TECHNICAL

A-1. ASSAY

A-1.0 Outline of the Method — 'IKA Universal Bomb' which is usually employed for micro, semi-micro and macro determination of halogens and sulphur in organic compounds, is made use of to disintegrate the organically bound bromine by fusion with sodium peroxide and ethylene glycol, thus converting it into the ionizable form. The mixture is then processed in the usual way and the halide determined potentiometrically by Volhard's method.

---

*Methods of sampling and tests for dye intermediates.

†Specification for water for general laboratory use (*second revision*).

A-1.1 Apparatus

A-1.1.1 *IKA Universal Shell Bomb*

A-1.1.2 *p*H *Meter*

A-1.1.3 *Silver Wire and Saturated Calomel Electrode*

A-1.2 Reagents

A-1.2.1 *Ethylene Glycol*

A-1.2.2 *Sodium Peroxide* — granules.

A-1.2.3 *Dilute Sulphuric Acid* — 50 percent (*v*/*v*).

A-1.2.4 *Standard Silver Nitrate Solution* — Approximately 0·1 M.

A-1.3 Procedure

A-1.3.1 *Determination of Total Bromine* — Weigh accurately 0·15 to 0·20 g of the sample, by difference, in the tared 'IKA' bomb. Add 7 to 8 drops of ethylene glycol using a dropping bottle provided with a small pipette fixed in a rubber stopper. The tip of the pipette shall be sized such that 8 drops are equivalent to 160 to 170 mg of ethylene glycol. Fill up the bomb with granular sodium peroxide starting little by little to avoid sudden reaction. The bomb shall be filled up, tapping it to settle the contents to 2 mm below the edge (about 11 g of sodium peroxide), care being taken so that no material is lodged between the lid and the rubber gasket. After the bomb has been filled, replace the lid and carefully close the bomb manually. Place the closed bomb in the housing provided in the safety jacket plate. The tip of the micro-flame shall barely touch the bottom of the bomb. Ignition shall occur after only 10 to 20 seconds (indicated by the red-hot colour at the bottom of the bomb). After a further 50 seconds, remove the bomb from the jacket with a pair of tongs and cool in water and open. The lid is thoroughly washed with water and washings collected in a 500-ml beaker. Boil the fused mass along with the bomb in water in the beaker covered with watch-glass for 15 minutes, and allow to cool. Acidify the solution with 25 ml of dilute sulphuric acid. Titrate this solution potentiometrically against silver nitrate solution using silver wire as indicator electrode and saturated calomel electrode as reference electrode. Note the potential electrode after addition of every 0·2 ml of silver-nitrate solution. The maximum potential difference indicates the end point. Continue the titration further till the difference in potential is reduced.

A-1.3.1.1 *Calculation*

$$A = \frac{V \times N \times 0{\cdot}079\,92}{M} \times 100$$

where

A = total bromine, percent by mass;
V = titre reading in ml;
N = normality of silver nitrate solution; and
M = mass in g of the material taken for the test.

A-1.3.2 *Determination of Free Halide* — Weigh accurately about 1 g of the dry material in a 250-ml beaker. Add 150 ml of water and heat to boil. Cool and add 2 drops of dilute sulphuric acid to make solution just acidic. Titrate the solution potentiometrically against silver nitrate solution as prescribed in **A-1.3.1**.

A-1.3.2.1 *Calculation*

$$B = \frac{V_1 \times N \times 0{\cdot}079\ 92}{M_1} \times 100$$

where

B = free halogen (as Br), percent by mass;
V_1 = titre reading in ml;
N = normality of silver nitrate solution; and
M_1 = mass in g of the material taken for the test.

A-1.4 Calculation

A-1.4.1 $D = A - (B + C)$

where

D = bromine due to bromamine acid, percent by mass;
A = total bromine, percent by mass (*see* **A-1.3.1.1**);
B = free halogen (as Br), percent by mass (*see* **A-1.3.2.1**); and
C = bromine due to 1-amino-2, 4-dibromoanthraquinone (*see* **A-2.4.2**).

A-1.4.2 Assay, percent by mass $= \dfrac{D \times 100}{20{\cdot}927}$

where

D = bromine due to bromamine acid (*see* **A-1.4.1**).

A-2. DETERMINATION OF UNSULPHATED MATTER

A-2.0 Outline of the Method — Unsulphated matter (1-aminoanthraquinone and 1-amino-2, 4-dibromoanthraquinone) is soluble in boiling toluene. It is continuously extracted by Soxhlet extraction and determined after removal of solvent.

A-2.1 Apparatus

A-2.1.1 *Soxhlet Extraction Unit*

A-2.1.2 *Round-Bottomed Flask* — 250 ml capacity with B 24 socket joint.

A-2.1.3 *Cellulose Thimble*

A-2.1.4 *Heating Mantle* — with control.

A-2.2 Reagents

A-2.2.1 *Toluene* — dry and freshly distilled.

A-2.3 Procedure

A-2.3.1 Weigh accurately about 30 to 35 g of the material and transfer to the thimble and place the thimble into the soxhlet extractor. Now weigh the round-bottomed flask with 2 or 3 glass beads. Add 150 ml of toluene and put the assembly together. Start heating at such a rate that there is a continuous steady trickle of hot solvent over the thimble. Continue the process of extraction till extract is colourless. Cool and remove the flask. Remove the solvent by distillation under reduced pressure preferably on a rotary evaporator. After complete removal of the solvent, dry the flask at 105 ± 5°C to constant mass in an electrically heated oven. Preserve the unsulphated matter for bromine estimation as indicated in **A-3.1**.

A-2.3.2 Determine the bromine content of the material as prescribed in **A-1.3.1**.

A-2.4 Calculation

A-2.4.1 Unsulphated matter, percent by mass $= \frac{m}{M_2} \times 100$

where

m = mass in g of the residue obtained after removal of toluene, and

M_2 = mass in g of the material taken for the test.

A-2.4.2 $C = \frac{V_2 \times N \times 0{\cdot}079\,92}{M_2} \times 100$

where

C = bromine due to 1-amino-2, 4 dibromoanthraquinone, percent by mass;

V_2 = titre reading in ml, calculated for the whole unsulphated matter;

N = normality of silver nitrate solution; and

M_2 = mass in g of the material taken for the determination of unsulphated matter.

A-3. DETERMINATION OF 1-AMINOANTHRAQUINONE-2-SULPHONIC ACID

A-3.0 Outline of the Method — 1-Aminoanthraquinone-2-sulphonic acid is determined by paper chromatography.

A-3.1 Apparatus

A-3.1.1 *Paper Chromatographic Chamber*

A-3.1.2 *Micropipette* — of 10 μl capacity.

A-3.2 Reagents

A-3.2.1 *Chromatographic paper* — in the form of a strip of size 25 cm × 16 cm.

A-3.2.2 1-*Aminoanthraquinone*-2-*sulphonic Acid* — pure (reference sample).

A-3.2.3 *Eluent* — a mixture of ethyl acetate, pyridine and water in the proportion 70 : 25 : 20 (by volume).

A-3.3 Procedure

A-3.3.1 Weigh accurately 0·5 g of the material and dissolve in 10 ml of a mixture of pyridine and water (in the proportion 1 : 1 by volume). Similarly prepare solutions of the reference sample of 0·5 percent, 1 percent, 2 percent and 3 percent concentrations. Take the chromatographic paper and mark a start line at a distance of 3 cm from any one end of the paper. Spot 10 microlitre each of the test solution and reference solutions along the start line at a distance of 3 cm from one another.

A-3.3.2 Now saturate the chromatographic chamber with the eluent by placing a trough containing the eluent at the bottom of the closed chamber. Suspend the chromatographic strip in the chamber in such a manner that the spotted end of the paper strip just dips into the trough containing the eluent. The eluent will travel upwards along the strip. Allow it to develop in this manner for 2 h during which time the solvent front travels approximately 15 cm past the start line. Now remove the strip from the chamber, dry it in air and observe under the normal light.

A-3.4 Reporting — Estimate the quantity of 1-aminoanthraquinone-2-sulphonic acid in bromamine acid by comparison of the intensity of colour of the spots of the material under test with those of the solutions of the reference sample.

A-4. DETERMINATION OF MOISTURE CONTENT

A-4.1 Determine the moisture content of the material as prescribed in **9.3** of IS : 5299-1969*.

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*Methods of sampling and tests for dye intermediates.

INDIAN STANDARDS

ON

DYE INTERMEDIATES

IS :	
2630-1975	Nitrobenzene (*first revision*)
2740-1973	Sulphanilic acid, technical (*first revision*)
2741-1973	β-Naphthol (*first revision*)
2744-1964	α-Naphthylamine
2833-1973	Aniline, technical (*first revision*)
3229-1973	Naphthionic acid (sodium salt) (*first revision*)
3242-1965	β-Oxynaphthoic acid (BON ACID)
3562-1965	*o*-Nitrotoluene, technical
4265-1975	4, 4′-Diaminostilbene-2, 2′-disulphonic acid (*first revision*)
4334-1967	*o*-Chloroaniline
4335-1967	*m*-Chloroaniline
4336-1967	*p*-Chloroaniline
4425-1967	*p*-Nitrotoluene-*o*-sulphonic acid
4523-1968	Acetoacetanilide
4524-1968	Acetoacet-*o*-chloroanilide
4525-1968	*p*-Aminoacetanilide
4526-1976	2, 5-Dichloroaniline (*first revision*)
4527-1968	2-Nitro-4-chlorotoluene
4528-1977	4, 4′-Dinitrostilbene-2, 2′-disulphonic acid (disodium salt)
5042-1976	1-Aminoanthraquinone (*first revision*)
5043-1976	2-Aminoanthraquinone (*first revision*)
5044-1969	Benzanthrone
5045-1976	Metanilic acid, technical (*first revision*)
5299-1969	Methods of sampling and tests for dye intermediates
5438-1977	Nitrobenzene-3-sulphonic acid, sodium salt
5646-1970	*p*-Anisidine
5647-1970	*p*-Toluidine
5648-1970	*o*-Anisidine
5649-1970	*o*-Toluidine
6258-1970	*o*-Nitroanisole
6259-1971	Anthraquinone, technical
6260-1971	*p*-Nitroanisole
6264-1971	J acid
6265-1971	Quinizarine, technical
6266-1971	1, 4-Diaminoanthraquinone, technical
6961-1973	3-Bromobenzanthrone, technical
6962-1973	3, 9-Dibromobenzanthrone, technical
6977-1973	1, 5-Diaminoanthraquinone, technical
7359-1974	1-Chloroanthraquinone, technical
7360-1974	1, 5-Dichloroanthraquinone, technical
7362-1974	Tobias acid
7364-1974	*m*-Nitro-*p*-toluidine
7635-1975	2-Nitroaniline
7636-1975	4-Chloro-2-toluidine
7637-1975	5-Chloro-2-toluidine
7641-1975	4-Chloro-2-anisidine
7642-1975	4-Aminophenol
7643-1975	4-Chloro-2-nitroaniline
7644-1975	3-Nitroaniline

7645-1975	Phenyl J acid, technical
7646-1975	Benzoyl J acid, technical
7647-1975	Acetoacet-2-toluidide
7661-1975	4-Aminoazobenzene
7686-1975	3 (N, N-diethyl) aminophenol
7704-1975	4-Aminotoluene-3-sulphonic acid
8059-1976	2-Chloro-5-aminotoluene-4-sulphonic acid
8111-1976	N, N-Dimethylaniline
8120-1976	Schaeffer's acid (sodium salt), technical
8193-1976	2-Nitro chlorobenzene
8194-1976	3-Nitro chlorobenzene
8195-1976	4-Nitro chlorobenzene
8196-1976	2, 4-Dinitrotoluene
8232-1976	2, 6-Diaminoanthraquinone
8233-1976	Phenyl peri acid
8397-1976	2-Nitrotoluene
8398-1976	3-Nitrotoluene
8399-1976	1, 3-Dinitrobenzene
8400-1976	2, 4-Dinitro chlorobenzene
8551-1977	1-(4′-sulphophenyl)-3-methyl-5-pyrazolone
8552-1977	1-(4′-sulphophenyl)-3-carboxy-5-pyrazolone
8553-1977	1-Phenyl-3-methyl-5-pyrazolone
8626-1977	R salt, technical
8627-1977	Gamma acid
8628-1977	G salt (Di-potassium salt), technical
8634-1977	Nitrodiazo acid
8637-1977	H acid

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	*Unit*	*Symbol*
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	*Unit*	*Symbol*
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	*Unit*	*Symbol*	*Definition*
Force	newton	N	1 N = 1 kg.m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Etectromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2